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(54) PROCESS FOR PROCESSING SEWAGE SLUDGE INTO THE FORM OF GRANULAR USABLE PRODUCTS

(71) We, CIBA-GEIGY AG, a Swiss body corporate, of Basle, Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The invention relates to a process for processing sewage sludge into the form of usable products; to the products obtained by the process; and to the use of these products.

There is described in German Offenlegungsschrift 25 20 528 (corresponding to British Specification 1513811) a process for isolating and drying solids from suspensions, which process comprises adding to the suspensions a liquid or a mixture of liquids, which wets to superficially dissolves the solids contained in the suspension and is insoluble or soluble to only a limited extent in the suspension liquid, in such a way that a liquid multiphase system is formed; subjecting the mixture to a thorough mixing operation until agglomerates of the solids have formed; and then separating these agglomerates from the liquid multiphase system and optionally drying them.

In a further study of this subject, it has now been found that application of the said process in a particular way to sewage sludge renders possible not only the isolation and granulation of the solids contained in the sewage sludge but also their conversion into usable products. This is due to the fact that there occurs, by virtue of the selection of suitable liquids, an extraction of, for example, noxious and odorous substances from the sewage sludge. This kind of extractive purification of sewage sludges, combined with an agglomeration of the solids in the sludge to form expanded granules, and the isolation and use of these, has not been known hitherto.

The present invention provides method of processing sewage sludge, which method comprises adding to the sewage sludge at least one liquid in an amount sufficient to form a multi-phase system and to agglomerate the sludge solids and optionally further additives; thoroughly mixing the multiphase system to extractively purify said sewage sludge and until the sludge solids form expanded agglomerated granules: and separating the resulting granules of solids from the liquid phase, and then drying the granules; with the proviso that in the case where the solids content in the sludge is from 0.1 to 35 per cent by weight said liquid is a mixture of isobutanol and methanol and in the case where the solids content in the sludge is above 35 per cent by weight said liquid is a mixture of isobutanol and methanol, or a hydrocarbon or halogenated hydrocarbon or a mixture thereof.

By "sewage sludge" is meant herein the sludge remaining behind in the settling tanks of purification plants after purification of domestic and industrial effluents. The different forms of sludge are distinguished as follows:

a) raw sludge: this contains the sediments and floating substances of the effluent in their natural state (e.g. faeces, paper and vegetable residues); this raw sludge accumulates in the settling tank after separation of the coarse material retained on the trash rack and of the sediments of the sand traps; it is dangerous hygienically on account of the presence of pathogenic germs, spores and worm eggs.

b) activated sludge: this is a sediment from the settling tank after the biological purifying stage; in the fresh condition it is almost odorless and consists for the greater part of micro organisms and residues of substances which are not decomposable or decomposable only with difficulty, so that it is bacteriologically and parasitologically unsafe; and

c) digested sludge: this a product from the anaerobic fermentation of raw sludge and/or activated sludge.

These sludges have approximately the following composition:

	Raw sludge %	Activated sludge %	Digested sludge %
fats and oils	7 - 35	5 - 12	3 - 17
pentosans	1	2	1,5
hemicellulose	3 - 5	about 7	1,6
cellulose	4 - 6		0,6
lignin	6 - 8		8,4
proteins	22 - 28	35 - 40	16 - 21
unknown substances	ca. 10	13 - 17	about 11
ash	20 - 40	25 - 35	40 - 55

Sewage sludge includes in a wider sense also the sludges of a general nature, which likewise can be processed by the process according to the invention. These are sludges from specific processes and/or the effluents therefrom, for example from municipal areas, e.g. from slaughterhouses, from food processing works and industrial plants, such as dairies, compressed yeast works and starch factories, from fermentation processes (penicillin works/streptomycin works, mycelium waste cultures, etc.), from breweries, and so forth; and likewise from agricultural works (e.g. processing of animal faeces into feeding stuffs).

The solids content in the sewage sludge is generally from 0.1 to 99 per cent by weight, particularly from 2 to 95 per cent by weight, and by sewage sludge is meant both the liquid sludge in the settling tanks and the dried sludge. Sludge liquids are both hydrophilic liquids, especially water, and hydrophobic liquids. The amount of liquid in the sludge is generally 99.9 to 1 per cent by weight, particularly from 5 to 98 per cent by weight.

It is possible by the process according to the invention to process both individual sludges and mixtures of these sludges.

Suitable halogenated hydrocarbons which may be used include, in particular, perchloroethylene, as well as 1,2-dichloroethane, chloroform and trichlorotrifluoroethane, and suitable hydrocarbons include hexane, and ligroin, for example petroleum ether.

It is also possible to use mixtures of solvents, for example 1,2-dichloroethylene of ethylene chloride with isopropanol.

Furthermore, other additives can be added in the process according to the invention. Suitable in this respect are, for example: solvent-soluble complexing agents, which serve to separate traces of metals, particularly traces of heavy metals; then products for fertilising or for conditioning the soil, such as urea-formaldehyde resins or commercial inorganic or organic fertilisers.

The process according to the invention comprises adding to the sewage sludge a suitable liquid as defined, optionally together with further additives, and subjecting the whole to a thorough mixing treatment, generally at a temperature of 15 to 200°C, preferably at room temperature, for, say, 1-60 minutes. The specified solvent systems form with the aqueous sewage sludge a multiphase mixture of at least one liquid phase and one solid phase, which on being stirred from agglomerated granules. The water has in the process the function of a bridging liquid which preferentially wets the solid material of the sludge. In order that the granulation can be carried out in a regulated manner, this bridging liquid has to be present at a suitably low concentration. Sewage sludges having solids contents of 0.1 to 35 per cent by weight thus have to be treated, before or during granulation, by means of the mixture of isobutanol and methanol.

During the granulating process, i.e. during the treatment of the sewage sludge with the solvents the biological cells present in the sewage sludge are forced open and consequently there are extracted, for example, the natural fats and oils, vitamins soluble in fats,

hydrocarbons that are difficult to break down, fetid substances from the putrefaction of proteins, and other lipidic substances and also water. At the same time, the solid material in the sludge swells up, with the volume during subsequent separation and drying being retained, and the porous structure being strengthened by intragranular bridges of solid matter.

After thorough mixing and granulation, the granulated solid material from the sludge is separated, for example by decanting, filtration, centrifuging and similar mechanical separation processes; the material obtained is subsequently dried by heating directly and/or indirectly.

After drying, the protein-containing biomass is in a purified, enriched and broken down form, and hence in a form readily usable for soil biology.

Using the process according to the invention, there is obtained a dry granulate from the sludge solids, which granulate usually has a particle size of 0.1 mm to 4.0 mm and the following specific properties:

- granulated, expanded, consolidated form;
- storage-stable at temperatures of −25°C to +50°C with relative humidity up to 95%;
- good pourability (noncaking);
- odourless;
- free from noxious substances;
- internal surface enlargement by about ×10 to ×150 as a result of swelling of the sludge solids (BET surface areas up to 35 m^2/g);
- porous structure;
- marked absorptive capacity (e.g. 200-300%, relative to its own weight, for hydrophilic liquids), as a consequence of which a specific water retention capacity and a correspondingly regulated release in dry soils are provided;
- good and rapid availability of nutritive substances (high protein content and favourable protein composition);
- bacteriologically and parasitically safe; and
- bulk density 0.5 to 0.15 kg/l.

By virtue of the fact that the process of the invention yields from sewage sludge, particularly because of the extraction of noxious substances, and optionally in consequence of the removal of heavy metals, a product which is incomparably more pure than that obtained from sewage sludge by the known thermal working-up processes, and renders possible in a simple manner the use of the said product, it is a process superior to known processes.

Owing to the specific properties mentioned, there are interesting industrial possibilities of application for the granulate obtained from the solids contained in sewage sludge. These "sludge solids granulates" can be used, for example, as fertilisers, effective soil conditioners, with suitable compatibility, as animal feedstuffs, impregnable animal feed additives, and also as carriers for active substances, e.g. as carriers for high-grade additives for feeding stuffs.

Besides the solids granulate, there is obtained a lipidic substance extract, which can be subjected to further utilisation or processing.

As a result of the application of granulates of the said kind in soil biology, there occurs, for example, an increase in the solid matter content (growth increase) of plants.

The following Examples further illustrate the invention without limiting the scope thereof. Temperature values are in degrees Centigrade. The internal surface area is measured according to the BET method (S. Brunauer, P. H. Emmet and E. Teller, J. Am. Chem. Soc., Vol. 60 (1958), pp. 309-319). The surface area of the granules is compared with the average surface area of the solid matter in the sludge having about 0.2 m^2/g (measured on solid matter from the sludge - dried by heating for 2 hours at 120°C).

Example 1

In a 5-litre beaker are introduced in the course of one minute at room temperature, whilst stirring with a glass-blade stirrer at about 400 r.p.m. is maintained, 2200 g of an isobutanol/methanol mixture (10:1) and 600 g of activated sludge (aqueous) containing 4.1% of solid matter. Granules are formed, and these are separated after 5 minutes from the filtrate by filtration through a suction filter. The moist granulate residue after drying is allowed to stand in the vacuum drying chamber until constant weight is attained. A grey odorless granulate having a particle-size distribution of <0.1 to >0.8 mm is obtained.

The granulates obtained have a specific surface area of 25 m^2/g (BET) and a bulk density of about 0.25 kg/l, and can be used as fertilisers, soil conditioners, carriers or animal feed additives.

Granulates having similarly good properties are obtained by using, instead of 2200 g of the mixture of isobutanol/methanol, 3000 g of n-butanol or 3000 g of isobutanol, with

otherwise the same working procedure.

Example 2

1.5 kg of a municipal activated sludge (aqueous) containing about 13.5% of solid matter is mixed at room temperature with 0.8 kg of an isobutanol/methanol mixture (10:1), and the mixture is subsequently pumped through a Fryma homogeniser Type MZ-50/R. The homogeneous mixture is added in a 10-litre beaker, with stirring at about 400 r.p.m., to 4.5 kg of the above-mentioned solvent mixture, and stirring is continued for 30 minutes. Filtration is subsequently performed by means of a suction filter and a cloth filter. The resulting granulate residue contains about 20% of water. This residue is then dried in a fluidised bed dryer, air dryer or vacuum dryer to yield 179 g of an odorless dry granulate.

The porous granulate obtained has a particle-size distribution of 0.1 to about 1.5 mm. The internal surface area is 21 m^2/g.

The resulting granulate formed from the solids in the sludge can be used according to the information given in Example 1.

Example 3

1.5 kg of a municipal activated sludge (aqueous) containing 13.5% of solid matter is granulated according to the instructions given in Example 2. The separated granules are firstly dried in air overnight on metal sheets, and subsequently kept in a vacuum drying chamber at 60° until constant weight is attained. There is obtained in this way a granulate (porous structure) having a particle-size distribution of about <0.1 to about 2.5 mm.

Examples 4 to 8

200 g of municipal activated sludge (aqueous) have a content of solid matter of 13.5% is granulated with different amounts of an isobutanol/methanol mixture according to Example 2, and dried in a vacuum drying chamber. The granulate properties as a function of the amount of solvent are given in the Table which follows. The columns contain the following values:

column 1: different amounts of solvent mixture: isobutanol/methanol (10:1) in parts by weight relative to 1 part of solid matter;
column 2: water content (%) of the moist granulate;
column 3: extracted lipidic substance fraction in % of the employed solid matter after concentration;
column 4: surface area in m^2/g of the granulate (granules);
column 5: bulk density in kg/l; and
column 6: mean granule size in mm.

TABLE

Example	1	2	3	4	5	6
4	18.5	59	9,1	5.8	0,42	1-4
5	20,4	37	9,8	30,4	0,22	0,5-1,6
6	22,2	23	10,3	31,2	0,22	0,2-1,2
7	29,6	14,8	13,2	36.7	0,16	0,2-1,2
8	37,0	9,6	13,4	37,0	0,16	0,2-0,8

The dried granules have a residual moisture content of below 5% of water. The extracted residue according to column 3 contains for the most part natural and mineral fats and oils.

Example 9

1200 g of municipal activated sludge (aqueous) containing 17-18% of solid matter is mixed at room temperature with 480 g of an isobutanol/methanol mixture (10:1); the mixture is then homogenised and put through a Fryma mill. The homogeneous mass is added, with stirring at 500 r.p.m., to 960 g of the same solvent mixture. After 1 minute there is added 960 g of the solvent mixture and, after a stirring time of 6 minutes, a further 1200 g of solvent mixture is added. As a result of the addition of the solvent mixture by stages, there is formed a granulate having a low proportion of dust. After about 20 minutes, filtration is performed by means of a suction filter, and the granulate is subsequently divided. The one half is dried in a vacuum drying chamber and the other half in a fluidised

bed dryer. There is no difference in the granulates obtained by the two methods of drying. The total amount of dry granules obtained is 162 g. The particle-size distribution of the resulting granulate is between <0.1 and about 1.5 mm. The surface area of the granuate (granules) is $\sim 27 m^2/g$. The granulates obtained have a firm porous structure, and are used as fertilisers, soil conditioners, carriers and animal feed additives.

Example 10

780 g of municipal activated sludge (aqueous) containing 17% of solid matter is homogenised together with 80 g of a urea-formaldehyde resin and 960 g of a mixture of isobutanol and methanol by means of a Fryma mill. There is then added dropwise in the beaker 220 ml of water in the course of 30 minutes, and by stirring are obtained large granules. To effect drying, a further 1920 g of an isobutanol/methanol mixture (~10:1) is added, and stirring is continued for 20 minutes; the solid products are filtered of with suction, and dried overnight at 60° in a vacuum drying chamber to yield about 180 g of granulate having a particle-size distribution of <0.1 to 4.0 mm. The surface area is 24.8 m^2/g. The granules have a consolidated porous structure, and produce in greenhouse tests after addition to maize crops a growth increase, relative to the solid substance of the maize, of 30 to 90% compared with that of untreated maize, and compared with the results from blank tests with urea-formaldehyde resin.

Example 11

350 g of a mixture of raw sludge and municipal activated sludge (about 1:1) (aqueous) having a total content of solid matter of 30% is mixed and homogenised with 210 g of isobutanol/methanol (10:1) by means of a Fryma mill at room temperature. There is then added in the beaker, with stirring, 188 g of the solvent mixture saturated with water, with the fine granules being built up within 5 to 15 minutes to larger granules (stirring rate about 800 r.p.m.). In order to dry the moist granules, 333 g of solvent mixture is added with a stirring rate of 300 r.p.m., and stirring is continued for about 3 minutes. The granules are separated by means of a suction filter, and are firstly dried in a fluidised bed dryer and then in a vacuum drying chamber. Dark hard granules which are resistant to abrasion are obtained. The particle size of the granulate obtained is between <0.1 mm and 4.0 mm. The surface area is 14.0 m^2/g. The granules have a porous structure and can be used according to the information given in Example 1. Granulates of equal quality are obtained by using, in place of 210 g of the isobutanol/methanol mixture, 400 g of cyclohexanone and, instead of the solvent mixture, likewise cyclohexanone, with the procedure otherwise being the same.

Example 12

12 kg of a mixture of raw sludge and activated sludge (aqueous) having a content of solid matter of 30% and 7.2 kg of a solvent mixture of isobutanol and methanol (10:1) are homogenised at room temperature in a Fryma mill. The homogenised mixture is transferred to a 100-litre glass vessel, and 8.4 kg of solvent mixture saturated with water is added with stirring. The build-up of the granules is finished after 20 minutes, and to effect drying there is added a further 19.2 kg of solvent mixture. Filtration with suction is performed after 5 minutes. Drying of the granulate is carried out in a fluidised bed dryer. The granules obtained (residual moisture 4.8%) are a little softer than those in Example 11 because more solvent had been used. The remaining details correspond to those given in Example 11.

Example 13

68.9 g of mixed sludge (about 47% content of solid matter) (aqueous) consisting of 50 g of a mixed sludge, comprising raw sludge and activated sludge, and 18.9 g of activated sludge dried by heating are homogenised with 225 g of perchloroethylene at room temperature by means of a Polytron stirrer. Small granules are formed during this stage. The material is then stirred in a beaker for 15 minutes at 600 r.p.m., in which time the build-up of granules commences. The granules are subsequently filtered off with suction. They are dried in a fluidised bed dryer and then in a vacuum chamber. The granulate obtained has a particle-size distribution of <0.1 mm to 4.0 mm. The mean surface area of the granules is 2.9 m^2/g. The granules have a porous structue and have good storage stability. They are used as fertilisers, soil conditioners, carriers or animal feed additives. Granulates of equal quality are obtained by using, instead of 225 g of perchloroethylene, an equal amount of hexane or 1,2-dichloroethane or petroleum ether or trichlorotrifluoroethane, with otherwise the same working procedure.

Example 14

1.5 kg of mycelium containing 35% of solid matter is suspended in a mixture of 2 kg of isobutanol and 340 g of methanol. Granules are subsequently built up during the addition of

475 g of water in the course of 25 minutes with vigorous stirring. The granulation process is brought to an end by the addition of a further 2 kg of isobutanol. Stirring is slowly continued for 15 minutes and filtration is subsequently performed. Drying of the granules in the fluidised bed dryer yields 420 g of dry granules. These have a particle size of <0.1 mm to 3 mm. The granules obtained have a porous structure and are used as animal feeding stuffs.

WHAT WE CLAIM IS:-

1. Process for granulating sewage sludge to form useful products, which process comprises adding to the sewage sludge at least one liquid in an amount sufficient to form a multiphase system and to agglomerate the sludge solids; vigorously mixing said multi-phase system until the sludge solids form expanded agglomerated granules; separating said expanded agglomerated granules from the liquid phase; and then drying the resulting granules; with the proviso that where the solids content in the sludge is from 0.1 to 35 per cent by weight, said liquid is a mixture of isobutanol and methanol, and when the solids content in the sludge is above 35 percent by weight said liquid is a mixture of isobutanol and methanol, or a hydrocarbon or a halogenated hydrocarbon or mixtures thereof.

2. Process according to claim 1, wherein the halogenated hydrocarbon used is perchloroethylene.

3. Process according to claim 1 or 2 which is performed at a temperature at 15° to 200°C.

4. Process according to claim 3 which is performed at room temperature.

5. Process according to any one of claims 1 to 8 wherein a complexing agent and/or a product for fertilising or soil conditioning is added to the sludge.

6. A process according to claim 1 substantially as described in any one of the foregoing Examples 1 to 14.

7. A granulate whenever obtained by a process as claimed in any one of claims 1 to 6.

8. A method of conditioning the soil, a fertiliser or feedstuff which comprises adding thereto a granulate as claimed in claim 7.

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